KPT 1090
PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Application of: Stephen W. Colley et al. Art Unit: 1797
Serial No.: 09/806,180
Filed: June 5, 2001
Confirmation No.: 5384
For: PROCESS
Examiner: Virginia Manoharan

July 7, 2008

TELEPHONE INTERVIEW SUMMARY

TO THE COMMISSIONER FOR PATENTS,

SIR:

The undersigned attorney thanks the Examiner for the courteous telephone interview conducted on May 28, 2008 and the Interview Summary prepared by the Examiner and mailed June 4, 2008. This Summary is intended to supplement the above-referenced Interview Summary prepared by the Examiner.

Rejections under 35 U.S.C. 112

It was agreed that the amendment to claim 14 made in Amendment E, filed April 2, 2008, overcomes the rejection of claim 14 under 35 U.S.C. §112, second paragraph, set forth in the Office action dated November 2, 2007.

Applicants' undersigned attorney maintained that claim 1, including the requirement of recovery of a first distillate containing no more than about 10 mol% water, adequately defines the subject matter that applicants regard as their invention such that further delineation of the process steps already specified therein is not required and that the rejection of claims 1-14 under 35 U.S.C. §112, second paragraph, should be withdrawn.

The Examiner indicated that she would take the undersigned attorney's comments into consideration, but did not indicate whether or not the rejection of claims 1-14 under 35 U.S.C. §112, second paragraph, would be withdrawn or maintained.

Rejection under 35 U.S.C. 103(a)

Applicants' undersigned attorney argued that the invention set forth in claims 1-14 is patentable over EP 0 151 886 with or without JP 5186392. In particular, the undersigned attorney pointed out that the requirement of step (d) in independent claim 1 of recovering a first distillate comprising ethyl acetate, ethanol, and not more than about 10 mol% water from the first distillation zone is an affirmative restriction that in part defines the manner in which the pressure swing distillation process is carried out and is neither shown nor suggested by the cited art. That is, the not more than about 10 mol% water limitation is a restriction that must be observed in the practice of the pressure swing distillation system to satisfactorily achieve the goal of the invention (i.e., purification of ethyl acetate from a feedstock comprising ethyl acetate, ethanol and water).

The Examiner asserted that the requirement in claim 1 that the first distillate recovered from the first distillation zone contain no more than about 10 mol% water is an obvious optimization of a process parameter. Applicant's undersigned attorney pointed out that the cited art does not establish that variation of the water content in the first distillate recovered from the first distillation zone is a result-effective variable, which is required for such a variable to be subject to optimization. The Examiner maintained that such teaching is found in the cited art, including at paragraph [0010] of the

English translation of JP 5186392, a copy of which was sent via facsimile by the Examiner to the undersigned attorney on May 28, * 2008 prior to the telephone interview and is attached.

No agreement regarding the allowance of the pending claims was reached during the telephone interview.

The Examiner proposed that an amendment to claim 1:

including the limitations of dependent claim 13;
removing the word "about"; and
including the requirement of "pressure swing distillation"

would potentially be useful overcoming the rejections of claims 1-14 under 35 U.S.C. §112, second paragraph, and 35 U.S.C. §103(a).

Applicants look forward to the next action on the merits and response on the record to the arguments set forth in Amendment E, filed April 2, 2008.

Respectfully submitted,

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VMK/sxm
* Attachment

Filed via EFS

JP,05-186392,A(1993)]

Japanese (PDF)

File Wrapper Information

FULL CONTENTS CLAIM + DETAILED DESCRIPTION TECHNICAL FIELD PRIOR ART EFFECT OF THE NVENTION TECHNICAL PROBLEM MEANS EXAMPLE DESCRIPTION OF DRAWINGS DRAWINGS

Translation done.]

Translated: 23:08:58 JST 05/28/2008
Dictionary: Last updated 04/11/2008 / Priority: 1. Chemistry / 2. Technical erm / 3. Biotechnology

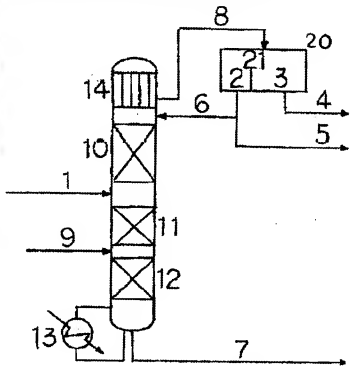
FULL CONTENTS

Claim(s)]

Claim 1] In the method of carrying out separation refinement of the ethyl acetate from the ethyl acetate which has ethyl alcohol in the concentration range within 45wt% to ethyl acetate, ethyl alcohol, and the mixture of water The out of the composition or the presentation near this which consists of the overhead with the above-mentioned mixture s made to distill using one distilling column. The refining nethod of the ethyl acetate characterized by obtaining ethyl lcohol and the ethyl acetate which hardly contains water rom the bottom of a distilling column when only the water-oor layer side whose most is ethyl acetate flows back to a listilling column, after condensing this mixture with a ondenser, and making the 2 liquid phase produced by ondensation separate.

Claim 2] The refining method of the ethyl acetate ccording to claim 1 characterized by keeping ethyl alcohol rom distilling from a bottom by carrying out addition

Drawing selection Representative draw



[Translation done.]

pouring of the water from the feeding stage of said distilling column at a lower distilling column stripping section.

Detailed Description of the Invention]

0001]

Industrial Application] This invention relates to the method of carrying out separation refinement of the ethyl acetate under existence of a catalyst from the ethyl acetate obtained by the synthetic reaction of ethylene and acetic acid, ethyl alcohol, and the mixture of water.

0002]

Description of the Prior Art] Although it is finishing (others Japanese Patent Application No. / 2-258233]) in patent application to make ethylene and acetic acid react under a gaseous phase by making a heteropolyacid into a main catalyst, and to manufacture ethyl acetate, about the refining methods of the resultant in an industrialization level, it is not yet solved. The water added for activity maintenance of a catalyst besides ethyl acetate and ethyl alcohol which is a by-product further are contained in said resultant. If the usual distillation operation tends to refine the mixture of these 3 yuan, an overhead steamy presentation forms ternary system composition, and since water and ethyl alcohol distill from a bottom with change of a feeding presentation, it cannot carry out separation refinement of the ethyl acetate from said mixture.

0003]

Problem to be solved by the invention] This invention tends to solve the above-mentioned problem and tends to offer the method of collecting high grade ethyl acetate efficiently.

0004]

Means for solving problem] [a distilling column stripping section / water] by carrying out addition pouring by this invention person forming a specific reflux system about the distilling method for the ability performing simultaneously dehydration and deethyl alcohol of ethyl acetate in one distilling column, as a result of repeating examination variously further It found out that the ethyl acetate of a high grade which does not contain water and ethyl acetate substantially was obtained from a distilling column bottom, and resulted in this invention.

0005] In the method of carrying out separation refinement of the ethyl acetate from the ethyl acetate with which the summary has ethyl alcohol in the concentration range within 15wt% to ethyl acetate, ethyl alcohol, and the mixture of water The cut of the composition or the presentation near this which consists of the overhead with the above-mentioned mixture is made to distill using one distilling

:olumn. After condensing this mixture with a condenser, nd making the 2 liquid phase produced by condensation :eparate, when only the water-poor layer side whose most is thyl acetate flows back to a distilling column Although it is he refining method of the ethyl acetate characterized by)btaining ethyl alcohol and the ethyl acetate which hardly :ontains water from the bottom of a distilling column, drawing 1 and drawing 2 explain the example of the :omposition of invention hereafter.

0006] Although the mixture of the ethyl acetate which has thyl alcohol in the concentration range not more than 45wt % from the feeding pipe 1 to ethyl acetate, ethyl alcohol, nd water tends to be supplied in drawing 1 , this tends to be listilled by a distilling column and it is going to collect the thyl acetate of the high grade through the bottom lischarging pipe 7 [in this case, ethyl alcohol in the)verhead distillate in the overhead discharging pipe 8] in)rder that it cannot condense and ethyl acetate may show the ction as a low-boiling point component to ethyl alcohol near / this / the presentation] more than [it is the above-nentioned ternary system composition] 8.4wt% -- the usual lowing-back method -- a column -- ethyl alcohol is nremovable from unbounded bleeding.

0007] In this invention, an overhead product separates into n after-cooling two phase, and moreover, paying attention o being higher than the ethyl alcohol concentration by the ide of a water-poor layer, like drawing 1 , the ethyl alcohol :oncentration by the side of a high-water-flow phase leads o a decanter 20 through the overhead distillate discharging)ipe 8, and carries out two-phase separation by a weir 21. nake the decanter high-water-flow phase 3 discharge hrough the high-water-flow phase discharging pipe 4 mong a two phase, and, on the other hand, the decanter vater-poor layer 2 leads the water-poor layer flowing-back)ipe 6 in a part -- a column -- it is made to flow back with he suitable reflux ratio for the upper part, and the remainder s made to discharge through the water-poor layer lischarging pipe 5 In flowing back of only a water-poor ayer, since it is large as compared with the case of the :oncentration of 3 yuan composition, even if the amount of lowing back of the rate of water concentration to the ethyl lcohol concentration in a water-poor layer of a water-poor ayer increases, it does not run short of water of 3 yuan equired for an azeotropy system presentation in the)verhead.

0008] Therefore, it results in change to the relative olatility of ethyl acetate and ethyl alcohol by flowing back)f only the water-poor layer flowing-back pipe 6, ethyl cetate comes to show the action as a high boiling point :omponent, and the ethyl acetate refined from the bottom is

)btained.
0009] [since an overhead part runs short of the moisture in
n azeotropy system presentation of 3 yuan, it will be
iecessary to compensate water with a certain method but, as
he water concentration in the 3 yuan mixture from the
eeding pipe 1 becomes low, and] a part of high-water-flow
hase 3 -- a column -- when it is made to flow back inside,
igh-concentration ethyl alcohol in the separated high-water-
low phase will be again returned to a column, and it is not
n effective method.
0010] Then, [in order to compensate the above and a water
hortage form the filling pipe 9 of water in a lower stripping
ection from the feeding stage, but] under the present
ircumstances, a column -- [with formation of a 3 yuan
zeotropy system by addition of water] to the case where
thyl alcohol has fallen to the stripping section according to
he turbulence of an inner delicate action It also has the
ffect of pulling up ethyl alcohol to an enriching section,
nd the injection rate of water installs the detecting element
f ethyl alcohol in a stripping section, and can control it by
eeding back detection concentration to the injection rate of
vater.
0011] About the presentation of a mixture, ethyl alcohol
oncentration to ethyl acetate 3 yuan 45wt%, namely, the
luality of ethyl alcohol and ethyl acetate -- by the above-
nentioned distillation method, when ethyl alcohol
xceeding an azeotropy system presentation is included,
ven if it carries out addition pouring of the water at a
listilling column stripping section, ethyl acetate is not
btained from a bottom, therefore ethyl acetate of 3 yuan
annot be refined from a mixture.
0012] [the presentation and temperature of an azeotropic
oint in the above-mentioned ternary system and the
rdinary pressure of a 2 element-system mixture] ethyl
cetate ethyl alcohol water (82.6wt% and 8.4wt% --) 9. 0Wt
%:70.2 Degree-C Ethyl Acetate Ethyl Alcohol (69.0Wt% --)
1. 0wt%:71.8 degree-C ethyl alcohol water (96.0wt% and
.0wt%): -- 78.2-degree-C water-ethyl acetate (91.5wt% and
3.5wt%): -- 70.4 degrees C is boiling point ethyl
cetate:76.8 degree-C ethyl alcohol:78.3 degree-C
vater:100.0 degree C of a pure substance again.
0013]
Working example]
Work example 1) The mixture of 3 yuan was refined using
he ORUDA show type rectifier with an inside diameter of
0mm which has the composition of drawing 1 . a column --
he upper part 10 -- 20 steps and a column -- the central part
1 -- ten steps and a column -- the mixed liquor (ethyl
cetate 94.6wt% and ethyl alcohol 2.0wt% and water 3.4wt
%) 100 weight part preheated from the feeding pipe 1 to 15

teps in the lower part 12 was supplied. Among the distillate rom the overhead, the presentation of the water-poor layer ? was ethyl acetate 87.2wt% and ethyl alcohol 6.1wt% and vater 6.7wt%, and when 117 weight parts were flowed back o the column through the water-poor layer flowing-back ipe 6 in this, the ethyl acetate which does not contain noisture and ethyl alcohol substantially was obtained from he bottom.

0014] (Work example 2) supplying the mixed liquor (ethyl cetate 94.3wt% and ethyl alcohol 3.3wt% and water 2.1wt %) 100 weight part preheated from the feeding pipe 1 using he same equipment as a work example 1 -- further -- a olumn -- the water 4 weight part preheated from the water illing pipe 9 of the stripping section was supplied. The resentation of a water-poor layer 2 among the distillate rom the overhead Ethyl acetate 87.7wt%, Ethyl alcohol 5.7wt%, it was water 6.5wt%, and when 173 weight parts were flowed back to the column through the water-poor ayer flowing-back pipe 6 in this, the ethyl acetate which loes not contain moisture and ethyl alcohol substantially vas obtained from the bottom.

0015]

Effect of the Invention] This invention enabled it to carry ut separation refinement of the ethyl acetate efficiently out f the mixture of the above-mentioned, the ethyl alcohol btained by the reaction path, water, and ethyl acetate.

Brief Description of the Drawings]

Drawing 1] a column -- it is the explanatory view having hown the concrete work example of this invention at the ime of using an inside condenser.

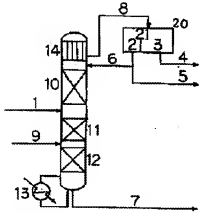
Drawing 2] a column -- it is the explanatory view having hown the concrete work example of this invention at the ime of using an outside condenser.

Explanations of letters or numerals]

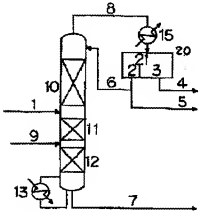
1 Feeding Pipe
2 Decanter Water-poor Layer
3 Decanter High-Water-Flow Phase
4 High-Water-Flow Phase Discharging Pipe
5 Water-poor Layer Discharging Pipe
6 Water-poor Layer Flowing-Back Pipe
7 Bottom Discharging Pipe
8 Overhead Discharging Pipe
9 Water Filling Pipe
10, 11, 12 Column
13 Reboiler
14 Overhead Condenser
15 External Condenser

20 Decanter
21 **

Drawing 1]



Drawing 2]



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